

High Performance Materials Applications to Moon/Mars Missions and Bases

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Abstract

Two classes of material processing scenarios will feature prominently in future interplanetary exploration: *in situ* production using locally available materials in lunar or planetary landings and high performance structural materials which carve out a set of properties for uniquely hostile space environments. To be competitive, high performance materials must typically offer orders of magnitude improvements in thermal conductivity or insulation, deliver high strength-to-weight ratios, or provide superior durability (low corrosion and/or ablative character, e.g., in heat shields). The space-related environmental parameters of high radiation flux, low weight, and superior reliability limits many typical aerospace materials to a short list comprising high performance alloys, nanocomposites and thin-layer metal laminates (Al-Cu, Al-Ag) with typical dimensions less than the Frank-Reed-type (e.g., packing flaws or 'weak' points crystallographically) dislocation source. Extremely light weight carbon-carbon composites and carbon aerogels will be presented as novel examples which define broadened material parameters, particularly owing to their extreme thermal insulation (R-32-64) and low densities ($<0.01 \text{ g/cm}^3$) approaching that of air itself. Even with these low-weight payload additions, rocket thrust

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limits and transport costs will always place a premium on assembling as much structural and life support resources upon interplanetary, lunar, or asteroid arrival. As an example, for *in situ* lunar glass manufacture, solar furnaces reaching 1700° C for pure silica glass manufacture *in situ* are compared with sol-gel technology and acid-leached ultrapure (<0.1% FeO) silica aerogel precursors.

Introduction

The Moon and Mars have a restricted, albeit plentiful, set of resources, and many materials will need to be imported from extralunar and extraplanetary sources for a long time. Because of this, anything not returning to Earth becomes a valuable source of raw materials for the lunar or Martian base. Lunar and planetary bases have been the subject of many design studies (Nishioka, et al. 1973; Carlton, et al., 1992; Dalton, et al. 1972; Mansfield, et al. 1971; Matsomoto, et al. 1997). In most cases, the total mass of housing and life support equipment is too large for efficient transport, reaching approximately 2000 t brought from Earth to accommodate a construction crew of 300 persons. *In situ* mining and processing is therefore a prerequisite for base construction and operation.

In various Moon or Martian exploration scenarios the costs of interplanetary transport become prohibitive in the absence of some *in situ* mining or production. The Mars Design Reference Mission (DRM) calls for the *in situ* production of CH₄/O₂ propellant for the crew's ascent vehicle and surface mobility, as well as the necessary water and life support gases for the crew's entire surface stay. *In situ* production permits a significant decrease in the amount of material that must be delivered to Mars from Earth, while simultaneously increasing the productivity of the crew on the surface and their safety. This paper surveys some of the high performance materials applications and attempts to assess advantages and disadvantages for Moon/Mars missions and bases, with particular attention paid to *in situ* production methods.

Material Abundance

Lunar Silica (SiO₂) composes approximately 40-50 percent of the lunar soil, and in abundance are also found oxides of aluminum (Al₂O₃), iron (FeO), magnesium (MgO), calcium (CaO), and titanium (TiO₂). Oxides of sodium (Na₂O), potassium (K₂O), phosphorus (P₂O₃), manganese (MnO), and chromium (Cr₂O₃) are present in less than 1 percent. There are about ten major oxides/silicates among lunar minerals. One of the most common lunar minerals is ilmenite, a mixture of iron, titanium, and oxygen. (Ilmenite also often contains other metals such as magnesium). Ilmenite is the most reactive phase, with complete reduction of FeO in the ilmenite to iron metal at all temperatures. Brecher et al. (1975) reacted lunar mare soil 74241 with hydrogen in a closed capsule at 800° C. They reported a significant increase in iron metal content at the expense of ilmenite. Electron micrographs showed a distinctive grain surface texture, composed of 1-5 μm metal sub-grains on a substrate of titanium oxide.

The most likely feedstock for a lunar oxygen plant is the local soil. Moon dust itself is a mixture of many different minerals, and nearly all of them contain oxygen in

considerable abundance. The Moon, even though it is 45 percent oxygen, is actually underoxidized. This is clear from the fact that the soil contains a high percentage of free iron (unoxidized) powder fines (harvestable for the cost of a magnet), and that oxidized iron (ore) is ferrous (FeO), not ferric (Fe_2O_3). About 85 percent of the weight of a typical spacecraft at launch is the oxygen used for rocket fuel. Lunar oxygen, condensed into liquid and stored in tanks made from lunar materials, might be shipped economically from the Moon to refuel spacecraft throughout cislunar space.

Base Geographic Consideration and Material Supply Lines Although still requiring confirmation, the Clementine satellite discovered lunar (south polar) ice—a potentially one billion metric ton supply of mainly water ice, but probably also containing propellant precursors such as frozen ammonia, methane, and other ices deposited by comets. Because the pole lies within the 2600-kilometer-wide, 12-kilometer-deep South Pole-Aitken Basin, the largest known impact crater in the solar system, the potential to partially cover a surface crater for habitation and base manufacturing is an appealing design. As a crater base, partial radiation shielding could be expected from the several million tons of soil mass passively and without impairing the contact of the colonists with their environment or imposing electromagnetic active shielding.

Some passive solar furnace made from lightweight reflectors is required for virtually all *in situ* production methods, particularly metallic ore or oxygen recovery (Mansfield, J. M., et al. 1973). For this reason, locating a base at the pole would furthermore allow the base to have portions in the Sun the majority of the time, especially mountain tops near the base. For lunar power generation, nuclear reactors have previously been considered a first choice compared to solar photovoltaics, since most places on the Moon receive 14 days of sunlight followed by 14 days of darkness. But at the south pole, near-constant sunlight shines at four places up to 90 percent of the time, making them excellent sites for solar power systems. The best-lit site is at 89.3 degrees south, 105 degrees west. Thus, placing two bases 'across' the lunar pole from each other would allow one of them to have sunlight at all times. The distance between them need not exceed 60 miles if by storage and transferring electricity from one base to the other, a dual site would allow incoming solar energy for photovoltaics or passive furnaces throughout the lunar night.

In situ Production and Extraction Cycles Lunar or Martian silica is needed for windows and solar cells. Aluminum, titanium, magnesium, and iron are all potential construction materials. On the Moon, titanium is in the form of a magnetic mineral (ilmenite) which can, in theory, be easily separated from the bulk of the lunar ore. In addition, use of titanium for structure would result in significant savings in the total amount of refined material because, although more difficult to form and fabricate, its strength-to-mass ratio is greater than that of the other metals available. Since ilmenite is basically FeTiO_3 , significant amounts of iron and oxygen can be extracted as byproducts. To supply one million tons per year, a surface area the size of about eight football fields must be mined. If the mining machinery operates 50 percent of the time, a mining rate of about 4 t/min (about 1 m³/min) is scooped and carried to the processor by scooper-loaders (Nishioka, et al. 1973), then it is carried from the mining area on a conveyor system.

Aluminum Apollo samples had an aluminum content between 4.5 and 14.4 percent. The Apollo missions did not provide any evidence of rich ore veins below the lunar surface. The various methods by which aluminum might be refined from lunar anorthosite include melt-quench-leach production of alumina followed by high temperature electro-winning of aluminum from aluminum chloride. The aluminum in lunar ore is in the form of plagioclase, $(\text{Ca},\text{Na})(\text{Al},\text{Si})_4\text{O}_8$, while magnesium and iron remaining after ilmenite removal are in the form of pyroxene, $(\text{Ca},\text{Fe},\text{Mg})_2\text{Si}_2\text{O}_6$. Because of the difficulty of economically separating the desired materials from association with such a wide variety of other elements, these are not normal sources of aluminum, magnesium, and iron on the Earth. Two processes, however, can be used to obtain alumina from anorthosite.

Anorthosite is a rock composed of plagioclase feldspar with minor amounts of pyroxene and olivine and is similar to the material found on the lunar surface. Detailed Explanation 1 (see end of article) shows some features of these processes. The direct production of metals by electrolysis of molten anorthosite is often proposed (e.g., see Nishioka, et al. 1973), but the results of research have been discouraging. The remaining possibility is the melt-quench-leach process which has succeeded in recovering over 95 percent of the alumina present in the ore. In this process, the ore is melted and then quenched to a glass, then treated with sulfuric acid to leach out the alumina component. Further treatment of the aluminum sulfate follows standard procedures that have been developed for low-grade bauxites and clays. Detailed Explanation 2 (see end of article) indicates some paths are possible once alumina has been obtained. The Hall Process is unsuitable because it would be extremely difficult to automate, and it consumes its electrodes and electrolytes. The subchloride process is much simplified, consisting of reacting aluminum chloride with alumina at high temperature to produce aluminum subchloride which later breaks down into aluminum and aluminum chloride. However, terrestrially, a pilot plant at Arvida, Quebec has reportedly shut down when the highly corrosive nature of the chloride vapor hindered scale-up. Of the two remaining processes, carbochlorination followed by reduction with manganese (Toth Process) is a batch process yielding a granular product which must be removed, melted, and cast. An extra carbon reduction process is also required. The high temperature electrolysis method is continuous and yields liquid aluminum ready for casting into ingots. The possibility of a melt-quench process (no leach) followed by direct extraction with a subchloride process, in theory, could reduce the plant mass by approximately 50 percent.

For forming metal composites of exceedingly high strength, Lehoczky (1978a,b) has reported that thin-layer metal laminates (Al-Cu, Al-Ag) with typical dimensions less than the Frank-Reed-type dislocation source have 2.4 times the tensile strength predicted by the rule of mixtures for similarly prepared metal specimens and 3 times greater than for cold-rolled bulk metals. In these Al-Cu laminates prepared by alternate vapor deposition, Cu is the minority species for lunar/Martian *in situ* production and may best be mined from meteor deposits or from scavenged parts of transport vehicles no longer needed on the surface.

Titanium The "baseline" process for oxygen and metal production involves reduction of ilmenite (FeTiO_3), a common mineral in lunar and terrestrial volcanic rocks.

The reaction, using hydrogen as a reducing agent, is: $\text{FeTiO}_3 + \text{H}_2 \rightarrow \text{Fe} + \text{TiO}_2 + \text{H}_2\text{O}$. Experiments using terrestrial ilmenite have demonstrated that it reacts completely at temperatures above 800° C in flowing hydrogen (Gibson et al., 1990). The reaction is fast at high temperatures, with reduction of the FeO complete in approximately 30 minutes at 1100° C (Allen et al., 1992a). In addition, TiO_2 is partially reduced to one of a series of suboxides with the general formula TiO_{2n-1} . In 3-hour experiments at 1100° C the dominant suboxide product was identified as Ti_4O_7 .

Rutile is titanium dioxide, the ore (TiO_2) commonly used for producing titanium metal on Earth. As a gemstone in its crystalline form or as powder in the most common white pigment used in paint. Most terrestrial titanium metal is produced from rutile laboriously mined from sands in Florida and Australia. However, if an efficient process is developed for extracting rutile from ilmenite, lunar materials research could have a positive effect on terrestrial production of titanium. For example, on Earth ilmenite is about fifty times more abundant than rutile. To separate ilmenite into its primary constituents, hydrogen is added to a heated mixture to produce raw iron, rutile, and water. Using a more complex process in a chlorine or fluorine reaction, the base can get pure titanium and more oxygen. Once the process is going, the hydrogen from water electrolysis can be recycled and used for the next load of ilmenite.

Oxygen Among the oldest and most technologically mature of the numerous lunar or Martian oxygen production methods which have been proposed, reaction of geologic materials in a reducing atmosphere at elevated temperatures converts FeO to iron metal and releases oxygen. The first oxygen production experiments to use actual lunar material were carried out by Carbotek (Knudsen et al., 1992; Ness et al., 1992; and Gibson et al., 1994). The experiments reacted lunar basalt 70035, crushed to <500 μm , with flowing hydrogen at 900 - 1050° C; JSC-1 contains 7.35 wt% FeO and 3.44 wt.% Fe_2O_3 (McKay et al., 1994). If all of this oxygen were released, the weight loss would be 2.67%. A 1 g sample reduced at 1050° C for 2 hours in the microbalance furnace lost 1.92 wt.%. Limited reduction of iron oxide in other common minerals has also been demonstrated (Massieon, 1992; Allen et al., 1993). Olivine, a common phase in many lunar samples, is partially reduced at temperatures around 1100° C. Smaller degrees of reduction have been documented in pyroxene, a major mineral in most lunar basalts and mare soils.

Glasses At least 25 distinct glass compositions exist in the Apollo sample collection (Delano 1986). The iron- and titanium-rich species, as represented by Apollo 17 orange glass 74220 (22.0 wt.% FeO, 8.8 wt. % TiO_2), promise particularly high oxygen yields. The orange glass deposit is uniformly fine grained and friable, offering a feedstock which reacts rapidly and can be used with little or no processing prior to reduction. Brecher et al. (1975) reduced a sample of Apollo 17 orange glass with hydrogen at 800° C in a closed reaction vessel. Oxygen yield, as measured by weight loss, increases as a function of FeO content. In all cases, significant portions of the iron oxide in the glass were converted to micrometer-scale iron metal blebs, with concomitant release of oxygen. At the same time, the glass devitrified to a fine-grained mixture of pyroxene and plagioclase crystals. Further melting of the glass between 1100 - 1125° C greatly reduces the surface area and decreases the reaction efficiency (Allen, et. al. 1992a-c). High surface

area silica aerogels have been proposed as rapid (nanoscale) catalysts and may offer advantages for extracting residual oxygen from processed glasses in late stages.

In addition to oxygen *in situ* production from glass oxides, glasses and silica aerogels can provide window areas for the lunar or Martian base structure. Silica (SiO_2), the basic ingredient in glassmaking, is found in abundance on the Moon. As a structural material glasses possess the desirable properties of low thermal expansion, high service temperature, good chemical, electrical, and dielectric resistance, and transparency to a wide range of wavelengths in the electromagnetic spectrum. To date, glasses made from lunar soil samples returned by the Apollo missions have been dark in color. The techniques necessary to manufacture glass from lunar materials which possess the properties needed for efficient transmission of sunlight into a lunar or Martian base have not been demonstrated; however, additional materials research will permit glass of adequate quality to be processed from the lunar/Martian soil with a minimum of additives (if any) brought from the Earth. Among important additives in terrestrial sheet glass, soda-lime glass is most commonly used. Its composition includes approximately 71-73 percent silica (SiO_2), 12-14 percent soda (Na_2O) and 10-12 percent lime (CaO). Soda is absent from the lunar soil in percentages needed for producing commercial soda-lime glass and it proves to be a costly item to supply from Earth. Fortunately, it does not appear to be necessary to supply additional Na_2O if a simple solar furnace for processing the lunar or Martian material is capable of generating temperatures considerably higher than those which could possibly be needed for this process, or if other oxides are substituted for soda-lime. Commonly used oxides in commercial glass which, if desired, would by necessity be additives to be mixed with the lunar materials, include lead oxide (PbO) used to provide X-ray and gamma-ray protection by absorption, and boric oxide (B_2O_3) used when good chemical resistance, high dielectric strength, and low thermal expansion are desirable.

One technique, which may prove feasible for large scale lunar/Martian *in situ* production, is the removing of almost all nonsilicate ingredients by leaching with acid. Again, the availability of high furnace temperatures is a prerequisite to meet the melting temperature of silica, and the manufacturing process will have to be shown to be manageable in space. The furnace temperature needed to melt pure silica (~ 1700 degrees C) is higher than that needed for soda-lime glass (~ 1550 degrees C) but is well within the limits of the solar furnace to be utilized. After additives (if any) are mixed with the lunar soil, the acid leaching stage removes undesirable materials from the mixture such as iron oxides which degrade the transmissivity of the glass. If it is decided to produce almost pure silica glass (> 95 percent SiO_2), almost all of the non-silicate constituents are leached out with acid at this stage. Requirements for providing these temperatures are calculated (Taylor, et al. 1992) as follows (1) 40 t/day maximum production schedule, (2) 24-hour work day, (3) Mean specific heat (0-1700 degrees C) ~ 1.13 J/g degrees C for silica glass, (4) Mean specific heat (0-1550 degrees C) ~ 1.21 J/g degrees C for soda-lime glass, and (5) Insulation 1.39 kW/m^2 . For silica glass, $\sim 890 \text{ kW}$ (640 m^2 solar collector) is needed. For soda-lime glass, $\sim 870 \text{ kW}$ (626 m^2 solar collector) is needed. A rough estimate for the weight of a plant processing 40 t/day is 400 t.

Silica aerogels Translucent aerogels can substitute for selected glass applications to insulate the lunar/Martian base against 400° F temperature swings in night/day cycles or

from the -30° F constant temperature near polar regions. As transparent, light weight insulators, this specialty material may offer substantial utility when derived from excess silicates in ore mining or silica glass manufacture. Aerogels can be made in silica, organic and pure carbon forms with the most useful densities generally being in the 0.08-0.15 g/cm³ range. On a lunar/Martian base, sodium silicate is a traditional terrestrial hydrolysis reaction leading to gel-based materials for air or vacuum drying into xerogels (higher density) or supercritical CO₂ drying into low density aerogels. The sol-gel method also allows aerogel to be molded into almost any shape and size (powder, beads, thin films, fibers, and monoliths). The thermal insulation characteristics of all the aerogels range from 3-10 times that of conventional organic foams or mineral wools with a fraction of the weight. When vacuum bagged (0.1 atm), R values are 20-36 per inch. In non-vacuum panels R values will be 10-15 per inch. Unique to aerogels is the extreme range of acoustic impedance (10³ to 10⁶ kg/m²s) and sound velocities (70 to 1300 m/s). This offers a new lightweight means of acoustic coupling or absorbing shock, having properties between those of a solid and a gas. All aerogels have extremely high surface areas; carbon versions have found applications in a new generation of supercapacitors, ion exchange media and lightweight composites. Unlike organic foams, silica and carbon aerogel are resistant to structural breakdown caused by solar radiation, radioactivity, and ozone. They offer stability to temperatures in excess of 500° C, with some forms of pyrolyzed carbon aerogels validated to as high as 2800° C, or 75 percent of the internal solar temperature. In particular, aerogel superinsulation is flexible cryogenic storage of liquid oxygen dewars for propellant and life support systems with extremely low thermal conductivity. The basic form of the super-insulation system is a blanket composed of aerogel-based radiation shield layers and low thermal conductivity aerogel/fiber matrix composites as a sheet, sleeve, or clamshell unit.

Carbon aerogels and composites Mars has permanent ice caps at both poles composed mostly of solid carbon dioxide ("dry ice"). The Sabatier Process, well known on Earth, follows the reaction $\text{CO}_2 + 4\text{H}_2 \rightarrow \text{CH}_4 + 2\text{H}_2\text{O}$. Water produced in the reaction would either be stored in the cache or dissociated to form hydrogen and oxygen. Oxygen would be stored in the fuel or life support cache, and hydrogen would be recycled into the Sabatier reactor. The only carbon or carbon products (e.g., formaldehyde) found in Apollo rocks is the trace put there by eons of buffeting of the surface by the solar wind. The atoms and ions from the wind are adsorbed to the surface of the fine particles of the regolith. Carbon is a trace only to be found in the upper meter or two where it amounts to 82 parts per million, and where it can be harvested by picking up top soil, then heating the surface deposits to 600 or 700 degrees Fahrenheit. If incorporated into polar ice, impact-derived carbon may deposit before volatilizing. It will be a major goal of future prospecting to find meteor-deposited surface metallic elements particularly for the otherwise scarce copper, zinc, etc.

Compared with metals, carbon matrix composites are two-thirds the weight of aluminum, and two and a half times as stiff. The coefficient of thermal expansion (CTE) of carbon composites is less than that of Invar. A CTE of zero can be tailored with medium to high modulus materials. Carbon/carbon (C/C) composites consist of carbon fibers in a carbon matrix and offer a repairable structural material with high strength and

low weight. Carbon composites are thermoformed at low processing temperatures ($\sim 300^{\circ}\text{F}$), but do require moderate carbonization temperature ($\sim 1000^{\circ}\text{F}$). C/C composites are often constructed by producing pre-forms of carbon fiber which are densified with carbon by either chemical vapor deposition or chemical vapor infiltration. Lunar soil can serve as cheap, expendable mandrels for molding shaped composites. These composites can withstand very high temperatures (e.g., above 2800°C) while maintaining strength and stiffness. Also, these composites are highly resistant to thermal shock and are stronger at high temperature (3000°F) than room temperature. C/C composites have been used in areas which require materials to withstand extreme variations in temperature and maintain some structural integrity. Some examples of uses of C/C composites from scavenged transport vehicles are: spacecraft brakes, engine pistons, space-shuttle leading edges and nose cones, and rocket nozzles and exhaust cones in remaining rocket stages that accompany the first landing and base establishment effort. After expending its fuel for the lower stage of the Apollo lunar lander, many potential scavenged precursors and metals were left on the surface for reuse or as additives in scarce structural materials.

Novel Assembly Methods In lunar (0.16 G) or Martian (0.33 G) gravity, and with a good vacuum, it may be practical to form a shell by using concentrated solar heat to melt aluminum or another metal at the center of a thin form. Evaporation over a period of months or years would build up on the form producing a metal shell for which the thickness at each point would be controlled by masking during the evaporation. This process would lend itself well to automation. Alternatively, or in addition, habitat sections could be constructed of fiber-composites. On Earth, the most familiar example of such a material is fiberglass--a mixture of glass threads in an organic matrix. Boron filaments are used in place of glass for high strength in aerospace applications. Glass fiber could easily be made from lunar materials. As a matrix, a silicon compound might be used in the space environment similar to a corresponding carbon-based organic. Such a compound might be attacked by the atmosphere if it were used on Earth, but could be quite stable in a vacuum.

Conclusions

New materials and novel production methods must be tailored both to weather the hostile lunar/Martian environment and, in some cases, allow for *in situ* production of structural and life support provisions. To support long duration missions to the Moon or Mars, breakthrough materials or propulsion methods are required which in both cases may depend on various "get it as you go" approaches. Aluminum, titanium and silica glass are likely structural materials with passive solar furnaces or photovoltaics providing energy in polar regions. Nuclear reactors are favored in many plans, but must be refueled from Earth over extended periods and carry high transport costs for items not easily repaired or serviced. The recent identification of potential frozen water at deep crater bottoms around the lunar southern pole and thin layer water deposits seasonally available in some analyses of dry ice Martian polar caps may change the way oxygen is generated. Oxygen is usually derived from heating soil and rock oxides, but with water available plans may now be made to establish crater bases with improved solar exposures and partial mass shielding of high dose radiation around crater walls.

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Detailed Explanations

1. Oxygen production chemistries.
2. Ilmenite leaching process description.

Explanation 1. Comparison between oxygen production chemistries

1. Hydrogen reduction of ilmenite only retrieves a small portion of the oxygen bonded in the lunar regolith. Ilmenite also only contains iron and titanium, so the scope of metal production as a spin-off is limited. 2. Hydrogen sulfide reduction yields greater oxygen than with the hydrogen reduction and H_2S is difficult to work with. 3. Carbothermic technique reduces only iron and titanium. Iron is only ~ 8-9% of the soil by weight. 4. Carbonate - oxygen reaction is quite complex, but has a low yield. 5. Fluorine exchange is an attractive process, which can extract all metals and oxygen. The metal fluorides can be sorted and processed. However, fluorine is notorious for being noxious to handle. As a result, if the oxygen is to be used for breathing, the oxygen must be very thoroughly cleansed. There is a possible source for fluorine from lunar apatite. Apatite also provides phosphorous, if phosphorous is needed for any purpose. 6. Chlorine exchange is somewhat similar to fluorine exchange, with the exception that chlorine only reacts with three or four of the oxides. 7. Electrolytic reduction with NaOH also extracts all of the oxygen. It also extracts all of the metals, albeit as a mixed slag.

Explanation 2. Ilmenite reduction for raw materials

The processing steps for reduction of ilmenite are as follows (1) Ilmenite is leached by hydrochloric acid solutions, or HCl vapor, to produce dissolved $FeCl_2$ and water, leaving a residue of TiO_2 and silicates, (2) Excess HCl and water are evaporated from the solution to increase the $FeCl_2$ concentration and adjust the pH, (3) Metallic iron is electrolyzed from the $FeCl_2$ solution and plated onto a suitable cathode. Chloride (or oxygen) is generated at the insoluble anode, (4) Excess water is recondensed and electrolyzed to yield hydrogen and oxygen. Alternatively, water and chlorine are reacted, using a suitable catalyst, to yield HCl and oxygen, (5) Hydrogen and chlorine are recombined by a suitable means, such as combustion or catalytic recombination, to yield HCl, (6) Regenerated HCl and sufficient water are recycled to leach additional ilmenite

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